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ELECTRON FLUX AT THE SURFACE OF TITANIUM TRITIDE FILMS

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Certain metal tritides have been investigated as reliable and quasi-constant sources of electrons for a number of practical purposes with particular attention to the dependence of the electron emission rate as a function of temperature. The objective of this article is to carry out simple calculations that illustrate the relative ranking of a number of binary metal tritides with respect to the maximum achievable electron flux; examine semi-empirically the energy spectrum of the electrons emanating from the surface of a titanium tritide film; and present experimental measurements of the electron emission rate from the surface of titanium tritide films. The results suggest that beryllium tritide would yield the greatest electron emission rate of all the metal tritides; the emitted flux has a significant component of secondary electrons; and, the total electron emission rate is quite sensitive to the condition of the emitting surface.

1. Introduction

The pure beta emitting character of tritium, its moderately energetic electrons of mean energy of 5.7 keV, and its relatively long half-life of 12.3 years has lent itself to a number of practical uses, some of which are as a primer for the RF discharge gap in plasma limiters in wave guides[1], an electron capture detector in gas chromatographs[2,3], a calibrator for ionization chambers[4], and quite extensively for the purpose of self-powered lighting[5]. Moreover, the ability of concentrating it in

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hydride[†] forming metals coupled with potentially very low equilibrium dissociation pressures at and near ambient temperature makes tritium a more tractable and desirable radioactive source of electrons. Several investigators[6,7,8] have examined the electron emission at the surfaces of partial tritides of titanium, yttrium, and scandium with particular interest in the rate of tritium desorption and electron emission rates as a function of temperature. In contrast, the objective of this study is to investigate the energy distribution of the electrons emanating from thin films of metal tritides having potentially the highest electron fluxes achievable. The work reported here, however, constitutes only the first instalment of this research, and in particular we consider the hydride forming metal titanium.

In the following section we present very plain arguments that serve to illustrate the relative ranking of metal tritides with respect to the maximum rate of electron emission. Subsequently, simple calculations are carried out to determine the electron energy distribution at metal tritide surfaces, and as an example the results for titanium tritide are given. In section 3 the experimental apparatus, the method of deposition and tritiation of titanium films, and the manner of measuring the electron emission rate are described. Lastly, the experimental results and discussion are presented in section 4. Measurements of the energy spectrum of the electron flux will be presented elsewhere.

2. Electron Emission and Energy Spectrum at the Surface of Metal Tritides

Energetic electrons traversing through matter undergo numerous electron-electron and radiative collisions in which they continually lose their energy and change their direction of propagation. At kinetic energies below the electron rest mass electrons lose energy principally via atomic or molecular excitation and ionization; a very small fraction is lost as bremsstrahlung and fluorescent radiation. At lower energies still electrons can suffer inelastic collisions that lead to relatively large deflections and relatively high energy losses. As a result of the combination of these collisional processes the electron trajectory through matter is highly tortuous and therefore the mean path length can be significantly greater (1.2 to 4 times)[9] than the mean range, where the latter is defined as the average distance travelled in a given direction. To obtain an approximate estimate of

[†] The term hydride is used in a generic sense, thus including all the isotopes of hydrogen.

the mean range of electrons with initial energy E through a given material, Bethe's expression[10] of the average energy loss per unit path length is integrated as follows:

$$\begin{aligned}
 R &= \int_0^R dx = \int_E^0 \frac{dx}{dE} dE = \int_0^E \left(-\frac{dE}{dx} \right)^{-1} dE \\
 &= \int_0^E \left(\frac{2\pi e^4 nZ}{E} \ln\left(\frac{4E}{I_{ave}}\right) \right)^{-1} dE \\
 &= (nZ)^{-1} f(E) \\
 &= \frac{\mathcal{A}}{\rho NZ} f(E). \quad (1)
 \end{aligned}$$

The above expression, which represents the average distance travelled by an electron in a given direction before coming to rest, is simply a product of the material properties (Z : atomic number; nZ : number of electrons per unit volume; ρ : material density; $\mathcal{A}$: atomic weight; N : Avogadro's number) and an energy dependent function $f(E)$ which is independent of the particular medium since the mean ionization and excitation potential I_{ave} is comparable for most materials. Using the empirical range-energy relationship for electrons of energies ~ 0.01 to 3 MeV in aluminium, the energy dependence can be explicitly included in the foregoing expression:

$$R = \frac{\mathcal{A}}{\rho Z} 0.20 E^{(1.265 - 0.0954 \ln E)} \quad (2)$$

R is in cm, ρ in g cm^{-3} , $\mathcal{A}$ in g mole^{-1} , and E in MeV.

In estimating the upper limit of the electron emission rate from a metal tritide film we assume that a film of thickness R , where R is evaluated at the maximum beta energy of 18.6 keV, is equivalent to an infinitely thick film in that betas produced at a depth greater than R will be absorbed in the medium and therefore not appear and contribute to the electron flux at the film surface. Furthermore, we assume that the film is of infinite lateral extent and electron propagation is equally probable in all directions, and therefore the maximum electron emission at the film surface is one half the rate of electron production in the bulk. The production and contribution of secondary electrons is not considered here. Thus, the electron flux ϕ at the surface can be simply expressed as:

$$\phi = \frac{1}{2} \frac{\rho_{MT_x}}{A_M + x A_T} x N \lambda R \quad (3)$$

where ρ_{MT_x} is the mass density of the metal tritide, A_M and A_T are the atomic weights of the hydride forming metal and atomic tritium, respectively, x is the tritium to metal atom ratio, λ is the probability of tritium decay, and R is the mean range of 18.6 keV

electrons in the metal tritide ($R = \frac{A_M + x A_T}{\rho_{MT_x} N (Z_M + x Z_T)} f(0.0186 \text{ MeV})$). Substituting

the expression for R in equation 3 we obtain the following simplified relation for the electron flux:

$$\phi = \frac{1}{2(Z_M + x Z_T)} x \lambda f(0.0186 \text{ MeV}). \quad (4)$$

The above expression indicates that the electron flux depends linearly on x , the tritium to metal atom ratio, and is inversely proportional to the weighted atomic numbers of the metal tritide. This suggests that hydride forming metals with low atomic numbers and large stoichiometric ratios will yield tritides with the highest electron emission rates. The electron flux for several metal tritides[†] along with other pertinent data is listed in Table I. At the top of the table we have included the electron emission from a "slab" of molecular tritium gas, which in the context of the above expressions can be thought of as a metal tritide of stoichiometry of 2; the metal in this case has an atomic weight and number of zero. This list shows that the maximum achievable electron flux is with tritium gas,

which is clearly a "tritide" with the largest ratio of $\frac{x}{(Z_M + x Z_T)} = \frac{2}{2 Z_T} = 1$. Of the metal

tritides we see that beryllium tritide has the highest electron emission flux followed by lithium and magnesium tritides.

[†] Only binary hydrides are considered.

Proceeding next to estimate the energy spectrum of the emanating electrons, consider an infinite and homogeneous metal tritide film of thickness t , mass density $\rho = \rho_{MT_2}$, and atomic weight $\mathcal{A} = \mathcal{A}_M + x \mathcal{A}_T$, as shown in Figure 1. Assuming that the tritium atoms, dispersed evenly in the material, decay uniformly, then the resulting primary^{††} electron energy distribution in any differential volume element $a \, dz$, where a is an arbitrary area and dz is the differential film thickness at depth z into the film, is given by:

$$\mathcal{N}_p(E) \, dE = \frac{x \mathcal{N} \rho}{\mathcal{A}} \lambda \mathcal{P}(E) \, dE \, a \, dz \quad (5)$$

where $\mathcal{N}_p(E) \, dE$ is the number of primary electrons in the energy interval dE about E , and $\mathcal{P}(E) \, dE$ is the probability of a tritium decay yielding an electron in the energy interval dE about E . Defining an energy and depth dependent transmission coefficient, $\mathcal{F}(z, E \rightarrow \hat{E})$, where $\mathcal{F}(z, E \rightarrow \hat{E}) \, d\hat{E}$ represents a fraction of electrons of initial energy E having an energy $\hat{E} \pm \frac{d\hat{E}}{2}$ after penetrating a distance z , and assuming that the tritium decay electrons are emitted uniformly in all directions so that on the average one half of the electrons are moving towards the $z=0$ surface, then the number of electrons, of initial energy $E \pm \frac{dE}{2}$, produced in a volume element $a \, dz$ at depth z , appearing at the $z=0$ surface with an energy $\hat{E} \pm \frac{d\hat{E}}{2}$ is given by:

$$\mathcal{N}_p(E) \, dE \, \mathcal{F}(z, E \rightarrow \hat{E}) \, d\hat{E} = \frac{1}{2} \frac{x \mathcal{N} \rho}{\mathcal{A}} \lambda \, a \, \mathcal{P}(E) \, \mathcal{F}(z, E \rightarrow \hat{E}) \, d\hat{E} \, dE \, dz. \quad (6)$$

Tacit in the definition of $\mathcal{F}$ is that the emerging electrons may not in fact be the primary electrons but that they are as a result of a multitude of collisional processes induced by the primary electrons; thus, it is an average representation of these processes. Integrating the preceding expression over the depth z and energy E (where E ranges from 0 to the

^{††} Unretarded tritium decay electrons produced in the volume element considered.

endpoint energy E_Q), and dividing by a yields an estimate of the energy dependent electron flux at the surface of a metal tritide film:

$$\phi(\hat{E})d\hat{E} = \int_0^{E_Q} \int_0^z \frac{1}{2} \frac{z N \rho}{\lambda} \mathcal{P}(E) \mathcal{F}(z, E \rightarrow \hat{E}) d\hat{E} dE dz. \quad (7)$$

The precision of the preceding expression is clearly hinged upon the accuracy of the energy-depth transmission coefficient $\mathcal{F}$. Fitting et al.[11,12] have published several studies in which they report an empirical expression for the energy-depth transmission coefficient of electrons through thin metallic foils of transmitted energies greater than 0.5 keV. In applying this data it is assumed that the transmission coefficient is not sensitive to whether the electrons originate from an external source, such as an electron gun, or are generated from within the bulk, as in the case of tritium decay. Fitting's data along with the tritium decay beta particle kinetic energy spectrum measured by Simpson[13] were used in the preceding equation to obtain the differential spectrum of the electron flux at the surface of titanium tritide films of various thicknesses. The energy spectra are shown in Figure 2 and certain integrated data are presented in Table II. The mode (or peak flux energy) of the energy spectra is seen to increase with increasing film thickness, a consequence of the form of $\mathcal{F}$, that is, negligible transmission for low energy electrons. The energy spectra show that the electron flux tends to saturate as the film thickness approaches a value of the order of 1 μm , as one would expect in view of the maximum range data obtained earlier. The spectra, however, do not show the presence of low energy electrons since Fitting's energy-depth relation considers only the higher energy electrons. In fact rigorous calculations would show a significant contribution at low energies, especially secondaries of ≤ 50 eV. In the tabulated data it is seen that as the film gets thicker a greater fraction of the decay electrons are absorbed in the bulk and as a result the fraction of decay electrons appearing at the surface diminishes markedly.

3. Experimental Apparatus and Procedure

An illustration of the experimental system is shown in Figure 3. The core of the experimental system consists of an ultra high vacuum (UHV) compatible 150 mm O.D. six-way cross with a substrate holder, a film deposition assembly, an electron collector,

gas inlet port connected to a 5 g uranium bed and Balzers HP (high pressure) gauge (with a range of 0.1×10^{-6} to 10×10^{-1} mbar), a viewport, and an UHV system on its six respective arms. The deposition assembly, electron collector, and the UHV pumping system can be isolated from the six-way cross by VAT gate valves. The substrate holder includes a stage which can be heated to 600°C and is outfitted with thermocouples and an electrical connector that can float the substrate potential with respect to ground. The deposition assembly, mounted on a linear motion device, can be positioned over the substrate within the six-way cross. The assembly includes high amperage copper feedthroughs and stainless steel rods to which hydride forming metal wires are spot welded. The feedthroughs and the rods are enclosed by a thin walled stainless steel shroud with an aperture of 38 mm in diameter which can be centrally positioned over the 63 mm diameter by 1.6 mm thick substrate. The distance between the plane of the titanium wires and the aperture is 13 mm while the distance from the aperture to the substrate is 16 mm. The electron collector, also mounted on a linear motion device, consists of a floating shield BNC connector (on a 70 mm O.D. Conflat flange) which is connected to a slender stainless steel rod with a 25 mm diameter electron collector disc at its end. The entire rod and the disc, except the collection face, are electrically insulated by a thin sleeve of borosilicate glass. A field defining stainless steel ring, electrically connected to the floating shield, is mounted around the collection disc, though electrically separated by the glass, so as to ensure that the field lines connecting the collector to the substrate cover an area no greater than that of the collector face. Therefore, the electron current reaching the collector is a measure of the electron emission rate. The gas and bypass lines are fabricated out of 10 mm I.D. electropolished 316 stainless steel tubing and fastened using VCR fittings; bellows sealed Nupro valves are used to achieve positive vacuum isolation. Tritium gas is stored on the uranium bed. The vacuum system consists of a Balzers TPU 170 turbomolecular pump, a Bayard-Alpert gauge, a Balzers QMG 112 quadrupole mass spectrometer, and a rotary vane backing pump. The experimental system proper is housed in a glovebox outfitted with all relevant tritium operation ancillaries. The glovebox environment is continually purged with a dry nitrogen atmosphere.

Typical titanium depositions are carried out using 0.5 mm diameter titanium wire of 99.99% purity spot welded to the stainless steel rods. Prior to the resistive heating evaporation of titanium the entire system is baked and rinsed in a hydrogen atmosphere at

temperatures of 150-200°C for a duration of ~2 weeks. The ultimate pressure achieved in the cross at ambient temperature (after a bakeout) is of the order of 10^{-9} mbar with the dominant specie being hydrogen. During deposition the average pressure in the vacuum chamber is less than 10^{-6} mbar. The total d.c. current draw by the 5 wires is ~26 amperes direct current at ~5 volts; the corresponding titanium wire temperature is ~1200°C. The duration of deposition is approximately 1 hour, resulting in an average deposition rate of $10 \mu\text{g cm}^{-2} \text{min}^{-1}$. Titanium films, which were subsequently charged with tritium, were deposited on stainless steel substrates. The thickness of the films was inferred from a calibrated quartz oscillator at a distance of 30 cm from the source. Glass substrates were used to determine the thickness distribution of the films by x-ray fluorescence (XRF); a traceable standard was used to calibrate the XRF. The films were relatively uniform in thickness, within 30% of the mean value, over a diameter of 40 mm; beyond this the thickness gradually diminished. Auger depth profiling of the untritiated films was carried out to determine its purity. Trace quantities of carbon, oxygen, and iron were detected; the source of iron was likely the stainless steel rods.

Following the evaporation of titanium, the deposition assembly, the electron collector, and the UHV pumping system were isolated prior to proceeding with the tritiation of the titanium film. Tritium was desorbed from the uranium bed into a calibrated volume to a pressure resulting in a charge of the order of a curie. The tritium gas was then expanded into the six-way cross and sorbed by the titanium film. This charging process was repeated until the film was loaded to close to stoichiometry. No change in the substrate temperature was detected during tritiding of titanium, the substrate being initially at room temperature. The remaining tritium was reabsorbed by the uranium and the chamber was reopened to the pumping system. The collector was then lowered towards the substrate to a separation distance of 2.5 mm. While ramping the potential of the substrate, negative with respect to ground, the current reaching the collector, which was held at a potential of a few mV above ground, was measured using a Keithley 616 electrometer. The collector current reached 93% of the limiting value at a substrate potential of -40 V; beyond -1.5 kV the current was essentially unchanged. The electronic energy emanating from the surface of the films was estimated by measuring, in an argon atmosphere, the saturation current at the collector. The product of the saturation current, which provides the number of ion pairs formed, and the average energy required

for the formation of each ion pair provides this estimate. Tacit in this discussion is that all the electronic energy is dissipated in producing the ion pairs.

4. Experimental Results and Discussion

Hydriding[†] of numerous evaporated titanium films of different thicknesses showed that films of thicknesses greater than about 150-200 nm suffered severe microcracking and exfoliation. During hydrogen charging immediately following a deposition it was observed that certain parts of the film which had experienced a greater thermal load from the hot titanium wires during evaporation did not undergo noticeable microcracking compared to those areas that were at lower temperatures. This suggests that the hydriding in the unaffected regions had occurred at a temperature where the internal stresses due to lattice expansion accompanied with the phase change (α (h.c.p.) to δ (f.c.c.)) were able to relieve themselves. In general, it was seen that hydriding of sufficiently thick titanium films invariably resulted in some microcracking and exfoliation, suggesting that the annealing of lattice expansion and transition stresses is quite sensitive to the temperature of the film, thermal gradients, and the rate of hydrogen charging. In order to readily overcome the difficulties of exfoliation of the titanium hydride films, the stainless steel substrate was sand blasted with 20 μ m glass beads prior to evaporation. Cracking of the hydrided film persisted, however the fracturing was now contained within the valleys created by the glass beading and thus the film did not exfoliate. Micrographs showing the roughened substrate and the hydrided titanium film are presented in Figure 4. Alternative techniques that show promise are: one, annealing of the deposited titanium film followed by hydrogen charging at temperature, thus relieving the accompanied lattice expansion and phase transition stresses; and two, evaporation of titanium in a hydrogen atmosphere which results in the growth of a series of atomic planes of titanium hydride leading to the formation of an integral film. Results of these tests will be presented elsewhere.

Tritium charging of titanium films proceeded in a manner not dissimilar to that of hydriding. The indicated pressure in the six-way cross, following volume expansion of

[†] Several titanium evaporation and hydrogen charging tests were carried out in a similar, non-active vacuum system to determine the appropriate conditions prior to proceeding with the experiment in the tritium system proper.

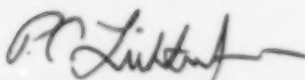
tritium, as a function of time for consecutive batch loadings of titanium film 1 is shown in Figure 5. The final indicated pressure in loadings 1 and 2 is significantly higher than that of subsequent loadings. Mass spectrometric analysis of the residual gas revealed a large peak at 3 amu, suggesting the evolution of the decay product of tritium, ^3He , from the partial uranium tritide lattice during tritium desorption.

Electron emission rate measurements along with other relevant data are shown for several titanium tritide films in Table III. The electron fluxes are approximately 10-15% of the maximum value for titanium given in Table I which shows the occurrence of significant absorption losses in the bulk. However, the measured electron flux (for the equivalently infinitely thick film 1) is larger than the computed value given in Table II. This difference can be attributed to the contribution of secondaries that was not included in the energy-depth transmission coefficient $\mathcal{T}$ used in the evaluation of equation 7.

The electron emission rate was observed to be quite sensitive to the condition of the emitting surface. For example, the electron flux from the surface of film 2, after several long duration (>1h) exposures to the glovebox atmosphere containing ~30 ppm of water, lead to a dramatic increase in the electron emission rate from a value of 0.22 nA cm^{-2} to 0.33 nA cm^{-2} . This increase, along with no measurable increase in the tritium concentration of the glovebox atmosphere upon removal of the substrate, suggests that the oxidation was primarily on the surface thus leading to an increase in the secondary yield. The slightly greater emission rate of film 3 than that of film 2, inspite of film 2 being thicker, is attributed to the poor vacuum during deposition; the ultimate pressure in the former case was $3 \times 10^{-8} \text{ mbar}$. In this case the increase in the secondary yield is likely due to the increased oxygen content in the bulk of the film as well as that on the surface.

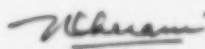
A saturation collector current of 42 nA cm^{-2} was measured for film 1 in an argon atmosphere; the collector-substrate potential difference was a 100 V. Using the W-value; that is the energy dissipation per ion pair formed for electrons traversing through argon, of 27.0 eV[14] yields an estimate of the electronic energy flux of $1.1 \text{ } \mu\text{W cm}^{-2}$. This value compares favourably with that presented in Table II.

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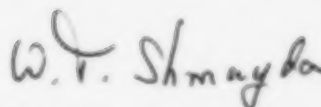


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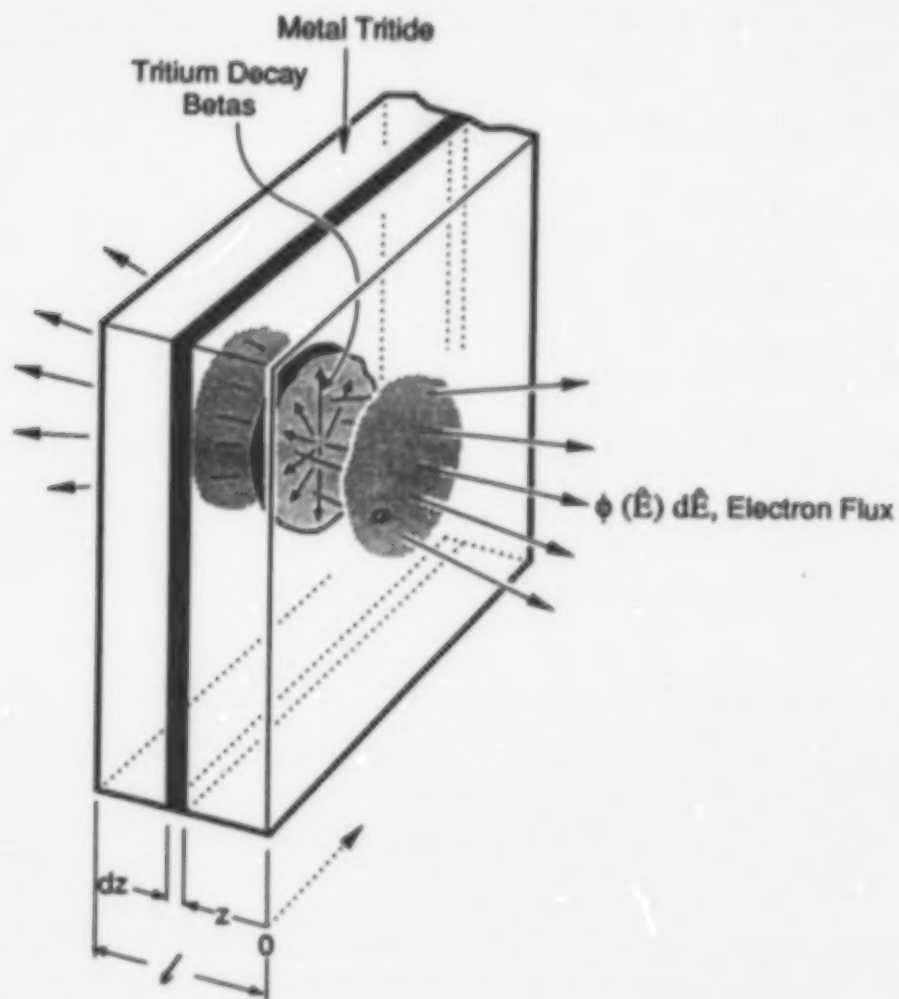


FIGURE 1
ELECTRON FLUX AT THE SURFACE OF A METAL TRITIDE FILM

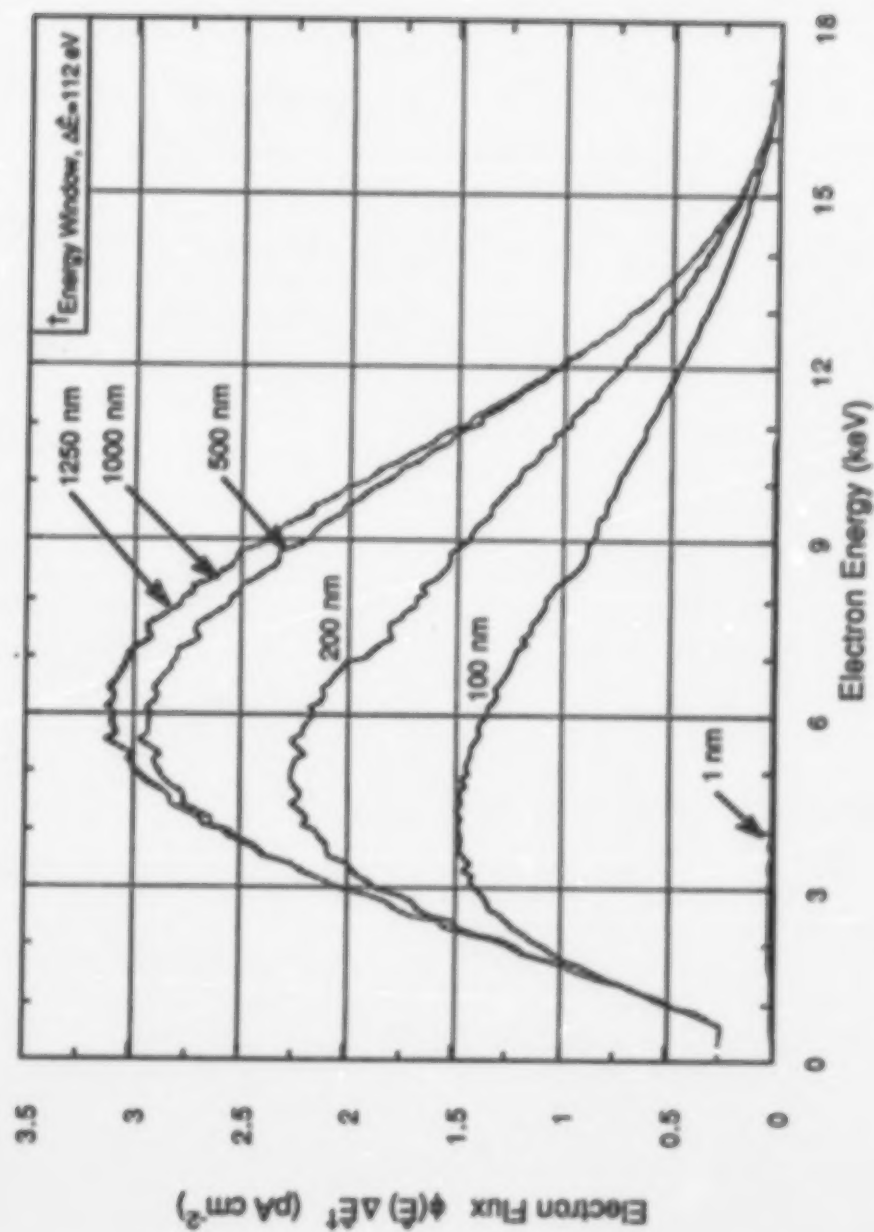


FIGURE 2
COMPUTED ENERGY SPECTRUM OF TRANSMITTED TRITIUM
DECAY BETAS ORIGINATING IN TITANIUM TRITIDE FILMS

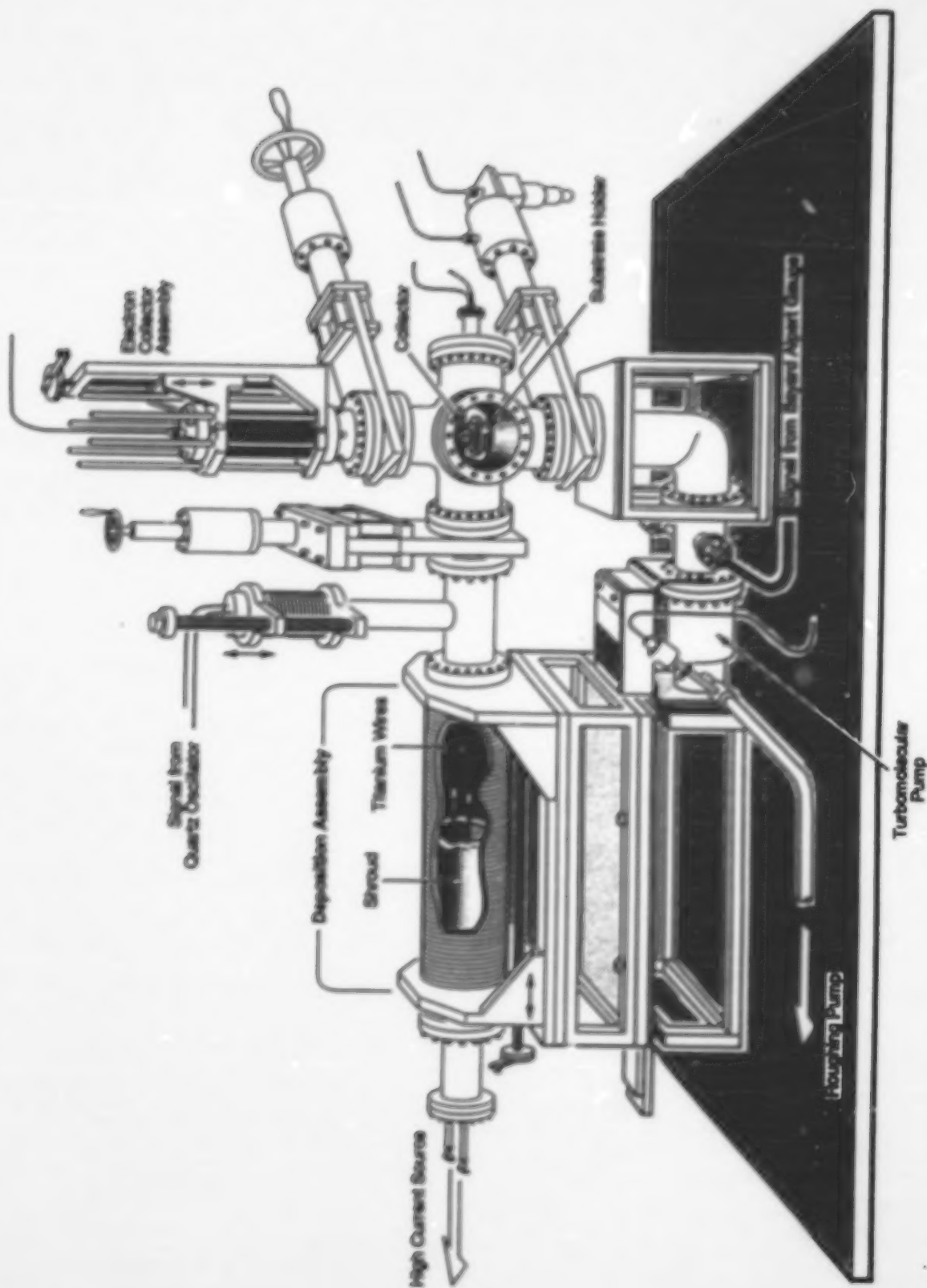
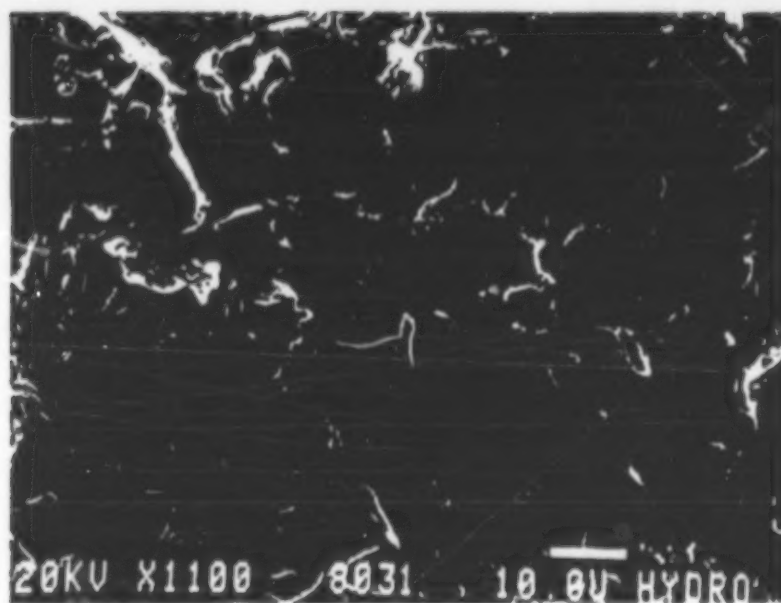


FIGURE 3
THIN FILMS DEPOSITION SYSTEM



a) Roughened Stainless Steel Substrate



b) Titanium Hydride on (a)

FIGURE 4

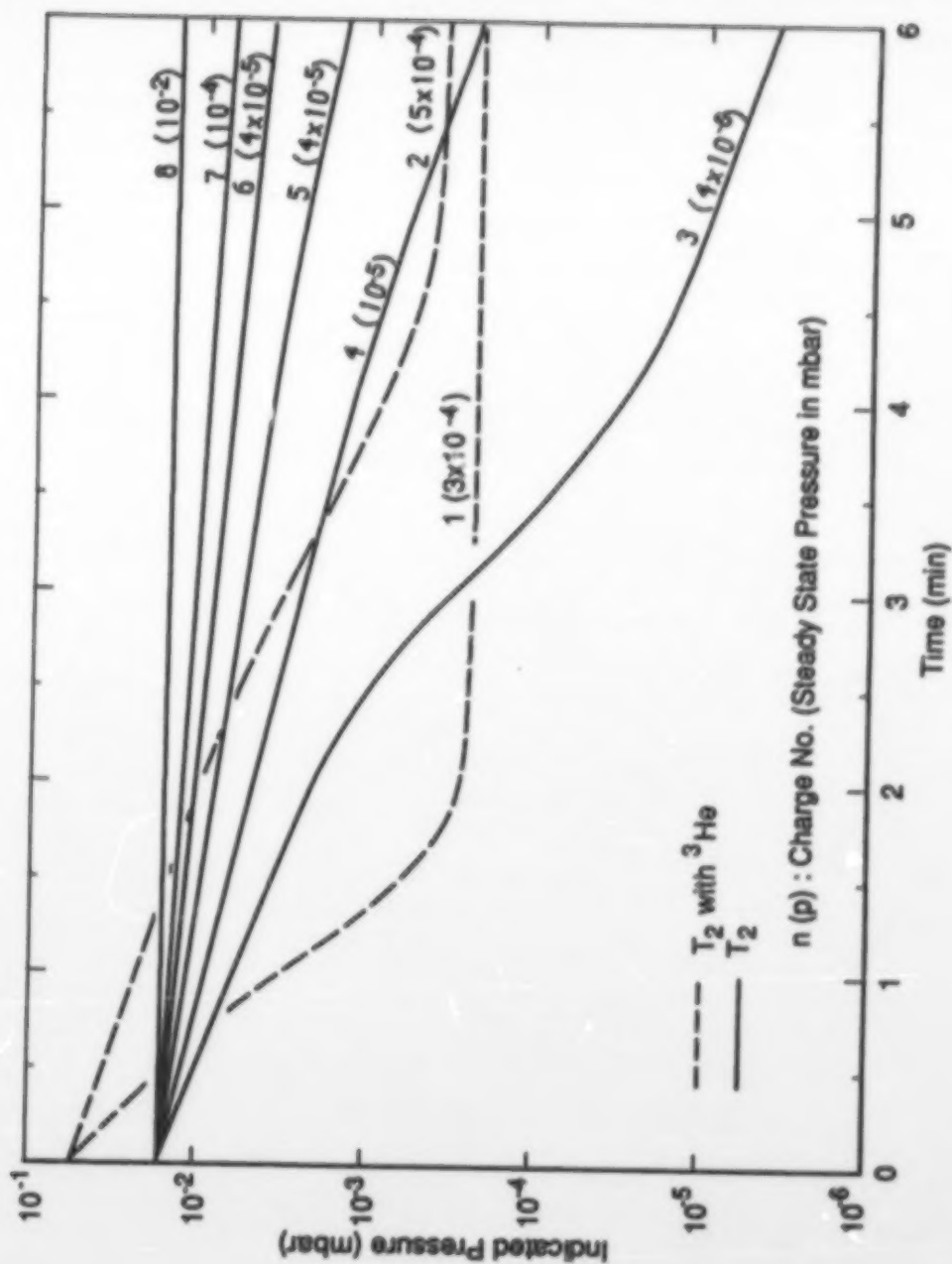


FIGURE 5

CONSECUTIVE TRITIUM CHARGING OF TITANIUM FILM 1
 (Charges 1 - 2 and 3 - 8 of 2.4 and 0.6 Ci, respectively)

TABLE I
ESTIMATED ELECTRON EMISSION RATE FROM METAL TRITIDE FILMS

Hydride Former	α	$\frac{\alpha}{Z_M + \alpha Z_T}$	Range [†] of 5.7 keV β (μm)	Range [†] of 18.6 keV β (μm)	Maximum Electron Flux (nA cm^{-2})
*	2	1.0	2.8×10^3	35×10^3	24
Li	1	0.25	1.1	13	6.1
Be	2	0.33	0.31	3.9	8.1
Na	1	0.083	0.51	6.4	2.0
Mg	2	0.14	0.28	3.5	3.5
K	1	0.050	0.56	7.0	1.2
Ca	2	0.091	0.31	3.8	2.2
Sc	2	0.087	0.17	2.1	2.1
Ti	2	0.083	0.11	1.4	2.0
V	1	0.042	0.088	1.1	1.0
Sr	2	0.050	0.20	2.6	1.2
Y	2	0.049	0.12	1.5	1.2
Zr	2	0.048	0.081	1.0	1.2
Ba	2	0.034	0.16	2.0	0.84
La	2	0.034	0.083	1.0	0.83
Hf	2	0.027	0.043	0.54	0.66
U	3	0.032	0.031	0.39	0.77

[†]Range is determined using the pure metal density at 300 K.

*Pure molecular tritium gas at normal temperature and pressure.

TABLE II
PARTICULARS OF THE COMPUTED ELECTRON FLUX
AT THE SURFACE^a OF TITANIUM TRITIDE FILMS

Film Thickness (nm)	Tritium Density (mCi cm ⁻²)	Electron Flux ^b (nA cm ⁻²)	Fractional Electron Output ^c	Electronic Energy Flux ^d (μW cm ⁻²)	Fractional Power Output ^e
1	0.54	0.003	0.50	0.02	0.50
100	54.5	0.12	0.36	0.74	0.39
200	109	0.17	0.27	1.15	0.30
500	272	0.23	0.14	1.57	0.16
1000	545	0.24	0.07	1.65	0.09
1250	681	0.24	0.06	1.65	0.07

^aThe flux and fractional quantities represent the output at one major surface of the film.

^b $\int \phi(E) dE$.

^c $\int \phi(E) dE / (\lambda * n_T)$ where n_T is the tritium atom density (per unit area).

^d $\int \phi(E) E dE$.

^e $\int \phi(E) E dE / (\lambda * n_T * 5.7 \text{ keV})$.

TABLE III
MEASURED ELECTRON FLUX AT THE SURFACE OF
TITANIUM TRITIDE FILMS

Film	Film Thickness			Electron Flux (nA cm ⁻²)
	(mg cm ⁻²)	(μm [†])	(μm ^{††})	
1	0.74	1.6	1.8	0.32
2	0.56	1.2	1.4	0.22
3	0.50	1.1	1.2	0.23

[†] Using solid tritium density of 4.5 g cm⁻³

^{††} Using titanium hydride density corrected for tritium: 4 g cm⁻³.